

# इंटरनेट

# मानक

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IS 12107-4 (1987): Methods of chemical analysis of alumino-silicate refractory materials, Part 4: Determination of phosphorus [MTD 13: Ores and Raw Materials]



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“Knowledge is such a treasure which cannot be stolen”



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## Indian Standard

METHODS OF  
CHEMICAL ANALYSIS OF ALUMINO SILICATE  
REFRACTORY MATERIALS

## PART 4 DETERMINATION OF PHOSPHORUS

**1. Scope** — This standard (Part 4) covers a method for determination of phosphorus (as  $P_2O_5$ ) in alumino silicate refractory materials.

**2. Determination of Phosphorus by Phosphomolybdate-Vanadate Complex (Spectrophotometric) Method**

**2.1 Outline of the Method** — An aliquot of the main solution is treated with ammonium vanadate in the presence of ammonium molybdate. Phosphomolybdate complex produced is measured at 450 nm in a spectrophotometer and amount of phosphorus pentoxide (as  $P_2O_5$ ) is evaluated from calibration curve obtained under similar conditions.

**2.2 Reagents**

**2.2.1 Concentrated nitric acid** (r.d. = 1.42) — Conforming to IS : 264-1976 'Specification for nitric acid (second revision)'.

**2.2.2 Dilute nitric acid** — 1 : 3 (v/v).

**2.2.3 Ammonium vanadate solution** — Dissolve 2.5 g of ammonium vanadate in water and dilute to 1 litre.

**2.2.4 Ammonium molybdate solution** — Dissolve 10 g of ammonium molybdate [ $(NH_4)_6MO_7O_{24} \cdot 4H_2O$ ] in 100 ml of water, filter if necessary.

**2.2.5 Standard phosphate solution** (1 g  $P_2O_5$ /litre) — Dissolve 1.917 g of potassium dihydrogen orthophosphate ( $KH_2PO_4$ ) in 1 litre volumetric flask and make up the volume. Take 100 ml of the solution and dilute to 1 000-ml in a volumetric flask (1 ml = 0.1 mg  $P_2O_5$ ).

**2.3 Procedure**

**2.3.1** Transfer two 50-ml aliquots of main solution [see 2.3.3 of IS : 12107 (Part 2) - 1987 Methods of chemical analysis of alumino silicate refractory materials: Part 2 Determination of silica] into two 250-ml beakers. Add 10 ml of concentrated nitric acid to each beaker and evaporate to dryness. Cool, dissolve each of the residue in 10 ml dilute nitric acid and transfer (filter, if necessary) the solutions to two 100-ml volumetric flask (A and B).

**2.3.2** To each flask, add 10 ml ammonium vanadate solution. To flask B only, add 5 ml ammonium molybdate solution. Dilute the solutions to the mark. Keep the solutions at room temperature for 10 minutes and measure the absorbance of solution B at 450 nm against the solution A.

**2.3.3 Calibration curve** — Draw a calibration curve, by taking 0, 1, 2, 3, 4 and 6 ml of standard phosphate solution (1 ml = 0.1 mg  $P_2O_5$ ) into six 100-ml volumetric flasks. Proceed in accordance with 2.3.2 and measure the absorbance of each solution at 450 nm against solution A. Plot the absorbance values against mg of  $P_2O_5$ /100 ml of the solution.

**2.4 Calculation** — Convert the photometric readings of the sample to mg of  $P_2O_5$  by means of calibration curve and calculate the  $P_2O_5$  content as follows:

$$\text{Phosphorus (as } P_2O_5 \text{), percent} = \frac{C}{D} \times \frac{1}{10}$$

where

C = mass in mg of phosphorus pentoxide ( $P_2O_5$ ) found in 50-ml aliquot of the final solution; and

D = mass in g of the sample represented by the 50-ml aliquot taken.

Adopted 24 August 1987

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## EXPLANATORY NOTE

Alumino silicate refractory materials contain alumina (  $\text{Al}_2\text{O}_3$  ) and silica (  $\text{SiO}_2$  ) in varying portions made synthetically by heating aluminium trifluoride at 1 000-1 200°C with silica and water vapour.

It is used in kilns, laddles and furnaces that operate at higher temperature or under conditions for which fireclay refractories are not suitable.

This Indian Standard has been prepared in different parts to cover the chemical analysis of various constituents in alumino silicate refractory materials. The other parts of the standard are:

- Part 1 Determination of loss on ignition
- Part 2 Determination of silica
- Part 3 Determination of aluminium
- Part 5 Determination of titanium
- Part 6 Determination of iron
- Part 7 Determination of manganese
- Part 8 Determination of calcium and magnesium
- Part 9 Determination of sodium and potassium.